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(54) Title: NONDIGESTIBLE FAT COMPOSITIONS CONTAINING SOLID POLYGLYCEROL ESTER PARTICLES FOR PASSIVE OIL LOSS CONTROL (57) Abstract Nondigestible fat compositions useful as a replacement for triglyceride fats or oils in foods are disclosed. The compositions have relatively flat Solid Fat Content (SFC) profile slopes between typical room and body temperatures. The nondigestible fat compositions comprise a liquid nondigestible oil and nondigestible solid polyglycerol ester particles dispersed in the oil in an amount sufficient to control passive oil loss. The ester groups of the solid polyglycerol ester particles comprise long chain (C ₁₆ -C ₂₄) fatty acid radicals, with at least about 40 % of the long chain fatty acid radicals being saturated and having at least 18 carbon atoms. Edible fat-containing products containing these nondigestible fat compositions can be less waxy tasting due to the lower level of solids required for passive oil loss control.		

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NONDIGESTIBLE FAT COMPOSITIONS CONTAINING
SOLID POLYGLYCEROL ESTER PARTICLES FOR
PASSIVE OIL LOSS CONTROL

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TECHNICAL FIELD

The present invention relates to nondigestible fat compositions that are useful as full or partial replacers for triglyceride fats or oils in foods. More particularly, the present invention provides such nondigestible fat compositions that provide passive oil loss control without being excessively waxy tasting.

BACKGROUND OF THE INVENTION

Certain polyol fatty acid polyesters have been suggested as low or reduced calorie substitutes for triglyceride fats and oils used in foods. For example, nonabsorbable, nondigestible sugar fatty acid esters or sugar alcohol fatty acid esters having at least 4 fatty acid ester groups with each fatty acid having from 8 to 22 carbon atoms have been used as partial or full fat replacers in low calorie food compositions. (See Mattson & Volpenhein; U.S. Patent 3,600,186; Issued August 17, 1971.) Foods in which these polyol polyesters are particularly useful as partial or complete replacements for triglyceride fats or oils include products suitable for use in frying. Unfortunately, regular ingestion of moderate to high levels of completely liquid forms of these polyol polyesters can produce undesirable passive oil loss, namely, leakage of the polyesters through the anal sphincter. By contrast, completely solid versions of these polyesters provide a sufficiently high solids content at mouth temperatures (e.g., 92°F, 33.3°C) such that they give a waxy taste or impression in the mouth when ingested.

As an alternative to these completely liquid or completely solid nondigestible/nonabsorbable polyol polyesters, certain intermediate melting polyol fatty acid polyesters have been developed that provide passive oil loss control, while at the same time reducing waxiness in the mouth. (See Bernhardt; European Patent Application Nos. 236,288 and 233,856; Published September 9, and August 26, 1987, respectively.) These intermediate melting polyol polyesters exhibit a unique rheology at body temperature by virtue of their having a matrix which involves a minimal level of solids (e.g. about 12% or lower) that bind the remaining liquid portion. As a result, these intermediate melting polyol polyesters are sufficiently viscous and have a suffi-

ciently high liquid/solid stability at body temperature to provide passive oil loss control. An example of such intermediate melting polyol polyesters are those obtained by substantially completely esterifying sucrose with a 55:45 mixture of fully hydrogenated (hardstock) and partially hydrogenated soybean oil fatty acid methyl esters. (See

5 Examples 1 and 2 of the above European patent applications.)

These intermediate melting polyol polyesters can be used as total or partial replacements for other fats and oils in various food products, including cooking and frying oils. However, it has been found that certain foods such as potato chips fried in frying fats containing substantial levels of these nondigestible intermediate melting polyol polyesters, particularly at levels in excess of about 40%, can give a significantly increased waxiness impression compared to potato chips that have been fried in the digestible triglyceride fat or oil that the nondigestible polyol polyester has partially replaced. (In terms of physical properties, "waxiness" relates to how the fat composition is sensed in the mouth, and specifically relates in part to the sensation of the product having a relatively high level of solids.) Indeed, this increased waxiness impression with regard to these intermediate melting polyol polyesters is recognized in the aforementioned European Patent Application No. 233,856 inasmuch as that application discloses fat compositions which contain digestible food materials, such as triglycerides and substituted mono- and diglycerides, that act as solvents for the intermediate melting polyol polyesters. However, as the proportion of triglycerides is increased relative to the intermediate melting polyol polyesters so as to impart less waxiness, the caloric content of the frying fat also increases accordingly. In addition, it has been found that frying fats containing greater than about 40% of these intermediate melting polyol polyesters can adversely affect the flavor display of the resulting fried food, in particular potato chips.

The waxiness impression imparted by intermediate melting polyol polyesters such as those of the aforementioned European '288 and '856 applications is believed to be due at least in part to their change in Solid Fat Content (SFC), particularly between typical room temperature (i.e. 70°F., 21.1°C.) and body temperature (i.e. 98.6o, 37°C.). For example, the intermediate melting sucrose polyester of Example 2 of European Patent Application Nos. 233,856 and 236,128 has an SFC profile slope (as hereinafter defined) between room temperature and body temperature of about -1.3. In other words,

the SFC profile slope of these intermediate melting polyol polyesters is relatively steep. Given this relatively steep SFC profile slope, the change in solids content of these intermediate melting polyol polyesters can be sufficiently great such that a high level of solids will be sensed when such room temperature materials are first placed in the mouth, thereby leading to an increased waxiness sensation.

5 Blends of completely liquid polyol polyesters with completely solid polyol polyester hardstocks, preferably esterified with C₁₀ - C₂₂ saturated fatty acids (e.g. sucrose octastearate), have also been proposed in order to provide passive oil loss control. (See, for example, Jandacek; U.S. Patent 4,005,195; and Jandacek/Mattson; 10 U.S. Patent 4,005,196; Both issued January 25, 1977.) Blends of these liquid polyol polyesters and solid polyol polyesters hardstocks have relatively flat SFC profile slopes between typical room temperature and body temperature, i.e. slopes of from 0 to about -0.3, and more typically from 0 to about -0.1. In other words, there is little or no change in the solids content of these blends between room temperature and body temperature.

15 Although providing at least temporary passive oil loss control, blends of liquid polyol polyesters and solid polyol polyester hardstocks according to the aforementioned U.S. '195 and '196 patents do not necessarily provide passive oil loss control over an extended period of time. It has been found that these solid polyol polyester hardstocks normally tend to form large spherulitic particles (typically from about 3 to about 32 20 microns in size) in the liquid polyol polyesters. These large spherulitic particles may tend to phase separate from the liquid polyol polyesters during storage of such blends. As a result, a two-phase system can develop with the liquid portion thereof providing minimal or no passive oil loss control.

In addition, blends of liquid polyol polyesters and solid polyol polyester 25 hardstocks according to the aforementioned U.S. Patents 4,005,195 and 4,005,196 do not necessarily lead to less waxy tasting products. As taught in these patents, a relatively high level of solid polyol polyester hardstock is required to provide passive oil loss control. For example, hardstock is preferably used in an amount of from about 20% to about 50% by weight of the liquid polyol polyester. (See Column 9, lines 65-68, 30 of U.S. Patent 4,005,195.) Such a level of solid polyol polyester hardstock used for passive oil loss control at body temperature can lead to a waxy tasting product due to the relatively high level of solids that will also be present at mouth temperature.

In view of the foregoing, it would be desirable to provide nondigestible fat compositions comprising blends of liquid polyol polyesters and solid polyol polyester hardstock particles with such blends exhibiting little or no phase separation of the hardstock particles from the liquid polyol polyesters. In addition, it would be desirable to be able to reduce the level of solid polyol polyester hardstock required for effective passive oil loss control so as to provide less waxy tasting products.

In addition to being useful as passive oil loss control agents when combined with liquid nondigestible oils, certain polyol polyesters which are solid at temperatures of about 25°C and higher have also been used as thickening agents for conventional digestible triglyceride oils. For example, these solid polyol polyesters have been used as "thickening agents" for blending with liquid digestible or nondigestible oils in formulations such as shortenings, as well as in other food products which contain a combination of fat and non-fat ingredients, e.g., margarines, mayonnaise, frozen dairy desserts and the like. (See, for example, Jandacek and Letton; U.S. Patent 4,797,300; Issued January 10, 1989.) However, these prior art thickening agents had to be used at levels of 10 to 25%. Accordingly, it would be desirable to reduce the level of thickening agents of this type in order to provide less waxy tasting products.

SUMMARY OF THE INVENTION

The present invention relates to nondigestible fat compositions which are useful as replacements for triglyceride fats and oils in food products. Such compositions have a Solids Fat Content (SFC) profile slope between room temperature (70°F) and body temperature (98.6°F) of from 0 to about -0.75 %solids/°F. Such compositions furthermore comprise a liquid nondigestible oil component having dispersed therein nondigestible solid polyglycerol ester particles in an amount sufficient to control passive oil loss upon the ingestion of the nondigestible fat compositions.

The liquid nondigestible oil component of the compositions herein is one which has a complete melting point below about 37°C. The polyglycerol esters which can be used to form the nondigestible solid polyglycerol ester particles used as oil loss control agents in compositions herein are those which have a complete melting point above about 37°C, wherein the ester groups therein comprise long chain (C₁₆-C₂₆) fatty acid radicals with at least about 40% of these long chain fatty acids having at least 18 carbon

atoms. The nondigestible solid polyglycerol ester particles dispersed in the liquid nondigestible component of the compositions herein can be further characterized as those which impart to the fat compositions herein a Thixotropic Area Value (as hereinafter defined) of at least about 10 kPa/sec.

5 The nondigestible fat compositions of the present invention provide significant advantages over known intermediate melting polyol polyesters, as well as prior art blends of liquid polyol polyesters and polyol polyester hardstocks. The relatively small nondigestible particles provide especially efficient passive oil loss control. As a result, the level of solids at body temperature required for passive oil loss control can be
10 reduced to relatively low levels (e.g., to less than about 20%, preferably to less than 15% of the nondigestible fat). In addition, the nondigestible fats of the present invention have relatively flat SFC profile slopes, thus leading to minimal or no change in the solids content between typical room and body temperature. This combination of the relatively low solids levels required for passive oil loss control, with minimal/no solids content
15 change between room and body temperatures, can result in less waxy tasting products containing these nondigestible fats.

 The present invention also relates to digestible fat compositions which utilize particles of the hereinbefore described nondigestible polyol polyester material as thickening agents. Such compositions comprise from about 85% to about 99% of a
20 digestible edible oil and from about 1% to about 15% of the nondigestible solid polyol polyester particles.

BRIEF DESCRIPTION OF THE DRAWINGS

Figure 1 is a template depicting the cooling profile of a potato chip.

25 Figure 2 is a photomicrograph (magnification of 1000X) depicting particles of solid polyglycerol ester dispersed in a liquid sucrose polyester.

DETAILED DESCRIPTION OF THE INVENTION

A. Definitions

30 By "nondigestible" is meant that only about 70% or less, preferably only 20% or less, more preferably 1% or less of a material can be hydrolyzed versus a triglyceride by the enzymes in the lipase test, described hereinafter in the Analytical Section.

As used herein, the term "thickness" of a particle is used in its conventional sense of the smallest of the three dimensions (length, width, height) of any given particle.

As used herein, the term "spherulitic" refers to substantially spherical or round, essentially three-dimensional particles.

5 As used herein, the term "platelet-like" refers to substantially flat, essentially two-dimensional type of particle having length and width in the unfolded planar configuration that is substantially greater in dimension than its thickness.

As used herein, the terms "filament-like" and "rod-like" refer to elongated, essentially one-dimensional particles.

10 As used herein, the term "complete melting point" refers to the temperature at which all solid components have melted. All melting points referred to herein are measured by Differential Scanning Calorimetry (DSC) as described hereinafter.

As used herein, the term "comprising" means various components, or steps, can be conjointly employed in the nondigestible fats, compositions, and processes of the present invention. Accordingly, the term "comprising" encompasses the more restrictive
15 terms "consisting essentially of" and "consisting of".

By "polyol" is meant a polyhydric alcohol containing at least 4, preferably from 4 to 12, more preferably from 4 to 8, most preferably from 6 to 8, hydroxyl groups. Polyols thus include sugars (i.e., monosaccharides, disaccharides and trisaccharides),
20 sugar alcohols (i.e., the reduction product of sugars wherein the aldehyde or ketone group has been reduced to an alcohol), other sugar derivatives (e.g., alkyl glycosides), polyglycerols such as diglycerol and triglycerol, pentaerythritol, and polyvinyl alcohols. Specific examples of suitable sugars, sugar alcohols and sugar derivatives include xylose, arabinose, ribose, xylitol, erythritol, glucose, methyl glucoside, mannose,
25 galactose, fructose, sorbitol, maltose, lactose, sucrose, raffinose, and maltotriose. Preferred polyols include erythritol, xylitol, sorbitol, and glucose, with sucrose being an especially preferred polyol.

By "polyol polyester" is meant a polyol as hereinbefore defined having at least 4 ester groups, i.e., at least 4 of the hydroxyl groups are esterified with fatty or other
30 organic acids. Polyol esters that contain 3 or less fatty acid ester groups are digested in (and the products of digestion are absorbed from) the intestinal tract much in the manner of ordinary triglyceride fats or oils, whereas those polyol esters which contain 4 or more

ester groups are substantially nondigestible and consequently nonabsorbable by the human body. It is not necessary that all of the hydroxyl groups of the polyol be esterified, but it is preferable that disaccharide molecules contain no more than 3 unesterified hydroxyl groups, and more preferably no more than 2 unesterified hydroxyl groups, so that they are rendered nondigestible. Typically, substantially all (e.g., at least about 85%) of the hydroxyl groups of the polyol are esterified. For liquid polyol polyesters, preferably at least about 95% of the hydroxyl groups of the polyol are esterified. In the case of sucrose polyesters, typically from about 7 to 8 of the hydroxyl groups of the polyol are esterified.

By "ester group" is meant a moiety formed from the reaction of a hydroxyl group with an organic acid or acid derivative which moiety contains fatty acid and/or other organic radicals having at least 2 carbon atoms, typically at least 8 carbon atoms, more typically at least 12 carbon atoms, most typically at least 16 carbon atoms. Representative examples of such fatty acid and other organic acid radicals include acetic, propionic, butyric, caprylic, capric, lauric, myristic, myristoleic, palmitic, palmitoleic, stearic, oleic, elaidic, ricinoleic, linoleic, linolenic, eleostearic, arachidic, arachidonic, behenic, lignoceric, erucic, and cerotic fatty acid radicals and other organic acid radicals including aromatic ester-forming radicals such as benzoic and toluic; branched chain radicals such as isobutyric, neooctanoic or methyl stearic; ultra-long chain saturated or unsaturated fatty acid radicals such as tricosanoic or tricosenoic; cyclic aliphatics such as cyclohexane carboxylic; and polymeric ester-forming radicals such as polyacrylic or dimer fatty acid. The fatty or other organic acid radicals can be derived from naturally occurring or synthetic acids. The acid radicals can be saturated or unsaturated, including positional or geometric isomers, e.g. cis- or trans-isomers, straight chain or branched chain aromatic or aliphatic, and can be the same for all ester groups, or can be mixtures of different acid radicals.

All percentages, ratios and proportions used herein are by weight unless otherwise specified.

B. Liquid Nondigestible Oil

A key component of the nondigestible fat composition herein is a liquid nondigestible oil having a complete melting point below about 37°C. Suitable liquid nondigestible edible oils for use herein include liquid polyol polyesters (see Jandacek;

U.S. Patent 4,005,195; Issued January 25, 1977); liquid esters of tricarballic acids (see Hamm; U.S. Patent 4,508,746; Issued April 2, 1985); liquid diesters of dicarboxylic acids such as derivatives of malonic and succinic acid (see Fulcher; U.S. Patent 4,582,927; Issued April 15, 1986); liquid triglycerides of alpha-branched chain carboxylic acids (see Whyte; U.S. Patent 3,579,548; Issued May 18, 1971); liquid ethers and ether esters containing the neopentyl moiety (see Minich; U.S. Patent 2,962,419; Issued Nov. 29, 1960); liquid fatty polyethers of polyglycerol (See Hunter et al; U.S. Patent 3,932,532; Issued Jan. 13, 1976); liquid alkyl glycosides fatty acid polyesters (see Meyer et al; U.S. Patent 4,840,815; Issued June 20, 1989); liquid polyesters of two ether linked hydroxypolycarboxylic acids (e.g., citric or isocitric acid) (see Huhn et al; U.S. Patent 4,888,195; Issued December 19, 1988); liquid esters of epoxide-extended polyols (see White et al; U.S. Patent 4,861,613; Issued August 29, 1989); as well as liquid polydimethyl siloxanes (e.g., Fluid Silicones available from Dow Corning). All of the foregoing patents relating to the liquid nondigestible oil component are incorporated herein by reference.

Preferred liquid nondigestible oils are the liquid polyol polyesters that comprise liquid sugar fatty acid polyesters, liquid sugar alcohol fatty acid polyesters, and mixtures thereof. The preferred sugars and sugar alcohols for preparing these liquid polyol polyesters include erythritol, xylitol, sorbitol, and glucose, with sucrose being especially preferred. The sugar or sugar alcohol starting materials for these liquid polyol polyesters are preferably esterified with fatty acids containing from 8 to 22 carbon atoms, and most preferably from 8 to 18 carbon atoms. Suitable naturally occurring sources of such fatty acids include corn oil fatty acids, cottonseed oil fatty acids, peanut oil fatty acids, soybean oil fatty acids, canola oil fatty acids (i.e. fatty acids derived from low erucic acid rapeseed oil), sunflower seed oil fatty acids, sesame seed oil fatty acids, safflower oil fatty acids, fractionated palm oil fatty acids, palm kernel oil fatty acids, coconut oil fatty acids, tallow fatty acids and lard fatty acids.

The nondigestible polyol fatty acid polyesters that are liquid are those which have minimal or no solids at body temperatures (i.e., 98.6°F, 37°C). These liquid polyol polyesters typically contain ester groups having a high proportion of C₁₂ or lower fatty acid radicals or else a high proportion of C₁₈ or higher unsaturated fatty acid radicals. In the case of those liquid polyol polyesters having high proportions of unsaturated C₁₈

or higher fatty acid radicals, at least about half of the fatty acids incorporated into the polyester molecule are typically unsaturated. Preferred unsaturated fatty acids in such liquid polyol polyesters are oleic acid, linoleic acid, and mixtures thereof.

The following are nonlimiting examples of specific liquid polyol polyesters suitable for use in the present invention: sucrose tetraoleate, sucrose pentaoleate, sucrose hexaoleate, sucrose heptaoleate, sucrose octaoleate, sucrose hepta- and octaesters of unsaturated soybean oil fatty acids, canola oil fatty acids, cottonseed oil fatty acids, corn oil fatty acids, peanut oil fatty acids, palm kernel oil fatty acids, or coconut oil fatty acids, glucose tetraoleate, the glucose tetraesters of coconut oil or unsaturated soybean oil fatty acids, the mannose tetraesters of mixed soybean oil fatty acids, the galactose tetraesters of oleic acid, the arabinose tetraesters of linoleic acid, xylose tetralinoleate, galactose pentaoleate, sorbitol tetraoleate, the sorbitol hexaesters of unsaturated soybean oil fatty acids, xylitol pentaoleate, and mixtures thereof.

The liquid polyol polyesters suitable for use in the compositions herein can be prepared by a variety of methods known to those skilled in the art. These methods include: transesterification of the polyol (i.e. sugar or sugar alcohol) with methyl, ethyl or glycerol fatty acid esters containing the desired acid radicals using a variety of catalysts; acylation of the polyol with a fatty acid chloride; acylation of the polyol with a fatty acid anhydride; and acylation of the polyol with the desired acid, per se. (See, for example, U.S. Patent Nos. 2,831,854, 3,600,186, 3,963,699, 4,517,360 and 4,518,772, all of which are incorporated by reference. These patents disclose suitable methods for preparing polyol fatty acid polyesters.)

C. Solid Polyglycerol Ester Component

A second key component of the nondigestible fat compositions herein comprises relatively small nondigestible solid particles of certain polyglycerol esters that are dispersed in the liquid nondigestible oil to control or prevent passive oil loss. These particles can be in a variety of forms and shapes, including spherulitic, platelet-like, filament-like or rod-like, or combinations of these various shapes, but are typically spherulitic or platelet-like. The thickness of these particles is typically about 1 micron or less. Thinner particles, however, are preferred from the standpoint of providing more efficient passive oil loss control of the liquid nondigestible oil component of the compositions herein. Accordingly, these particles preferably have a thickness of about

0.1 micron or less, more preferably about 0.05 micron or less. These particles also have a complete melting point of above about 37°C, preferably above about 50°C, more preferably above about 60°C.

The polyol polyester material which forms these nondigestible particles should
5 have a complete melting point as measured by the Differential Scanning Calorimetry (DSC) described hereinafter in the Analytical Methods section which is sufficiently high such that the nondigestible particles themselves will have the hereinbefore specified melting point characteristics when such particles are dispersed in the liquid nondigestible oil. For example, a polyol polyester material having a complete melting point right at 37
10 °C may not form solid particles having a complete melting point above about 37°C when such particles are dispersed in the liquid nondigestible oil. Thus, in some cases, the complete melting point of the neat polyol polyester material may have to be slightly higher than 37°C, e.g., about 40°C or higher, in order to form solid particles having a complete melting point of 37°C when such particles are combined with the liquid
15 nondigestible oil.

These nondigestible particles can generally be dispersed as discrete, unaggregated entities in the liquid nondigestible oil. However, these nondigestible particles can also cluster together to form much larger aggregates which are dispersed in the liquid nondigestible oil. This is particularly true of those nondigestible particles that
20 are platelet-like in form. Aggregates of platelet-like nondigestible particles typically assume a spherulitic shape that is porous in character and thus capable of entrapping significant amounts of liquid nondigestible oil. It is believed that this porous structure and its concomitant ability to entrap large amounts of liquid nondigestible oil is why these aggregated, platelet-like particles, while not as efficient as the particles in
25 unaggregated form, can provide very effective and efficient passive oil loss control.

The polyglycerol esters used to form the fat compositions of the present invention contain at least about 2 glycerol moieties, more preferably from about 3 to 10 glycerol moieties, even more preferably from 4 to 8 glycerol moieties, and most preferably from 4 to 6 glycerol moieties. Typically mixtures of polyglycerol esters are
30 employed have an average degree of glycerine polymerization (\bar{n}) as hereinafter defined in the Analytical Methods section of from about 2 to 10, preferably from about 3 to 8, more preferably from about 3 to 6. The distribution of the number of glycerol

moieties in such polyglycerol ester mixture may be narrow or broad. Typically, at least about 30% of the hydroxyl groups of the polyglycerol esters are esterified with fatty acids. Preferably at least about 50% of the hydroxyl groups are esterified. The percent esterification of the polyglycerol ester material used herein can be determined in the manner set forth hereinafter in the Analytical Methods section.

The ester groups which form the solid polyglycerol ester component herein comprise long chain (C_{16} - C_{26}) fatty acid radicals with at least 40% of these long chain fatty acids being saturated and having at least 18 carbon atoms. Preferably, at least about 50% of the long chain fatty acids are saturated and have at least 18 carbon atoms, more preferably at least about 75% of the long chain fatty acids are saturated have at least 18 carbon atoms, most preferably at least about 85% of the long chain fatty acids are saturated have at least 18 carbon atoms.

The fatty acid radicals forming the ester groups on the polyglycerol ester component herein may be saturated or unsaturated. The polyglycerol ester component can, in fact, be further characterized by specifying an Iodine Value which is a measure of the degree of unsaturation of the fatty acids which form the ester groups. The solid polyglycerol esters of this invention preferably have an Iodine Value of less than 50, preferably less than about 20, more preferably less than about 10, and most preferably less than about 5.

Mixed fatty acids from source oils (e.g., soybean oil, cottonseed oil, safflower, rapeseed oil, canola, corn oil, sunflower oil, and tallow) which contain the desired fatty acids can be used to form the fatty acid radicals of the ester groups of the polyglycerol ester materials used herein. For example, hardened (i.e., hydrogenated) high erucic rapeseed oil fatty acids can be used instead of pure behenic fatty acid. The fatty acids can be used "as is" and/or after hydrogenation, and/or isomerization, and/or purification. Preferably, the behenic acid (or its derivatives-e.g., methyl esters) are concentrated, for example, by distillation.

The solid polyglycerol ester materials used herein can be made according to known methods for preparing polyol polyesters. One such method of preparation comprises reacting the acid chlorides or acid anhydrides of the desired ester-forming acids, or the acids per se, with polyglycerol. This can be accomplished using a sequential esterification process or a process in which all the fatty acids are mixed

together and added at once. See Letton; European Patent 311,154; published April 12, 1989 (herein incorporated by reference).

Another method for preparing these solid polyglycerol esters is by a process which comprises reacting the methyl esters of the respective desired acids with polyglycerol in the presence of a fatty acid soap and a basic catalyst such as potassium carbonate. See, for example, Rizzi et al; U.S. Patent 3,963,699; issued June 15, 1976; Volpenhein; U.S. Patent 4,518,772; Issued May 21, 1985; and Volpenhein; U.S. Patent 4,517,360; Issued May 14, 1985, all of which relate to polyol polyester synthesis and which are incorporated by reference.

10 D. Preparation of Nondigestible Fat Compositions Which Exhibit Minimal Passive Oil Loss

To prepare the nondigestible fat compositions herein which exhibit improved passive oil loss control, the liquid nondigestible oil is combined with the particles of the solid polyglycerol esters hereinbefore described. The polyglycerol ester particles are used in an amount sufficient to control or prevent passive oil loss. What constitutes "an amount sufficient to control or prevent passive oil loss" for any given fat composition depends on the particular polyglycerol esters utilized therein, the particular passive oil loss control benefits desired, and the level of waxiness mouth impression that can be tolerated for the particular end product use of the nondigestible fat composition which is formulated. Typically, the nondigestible fat composition so formed will comprise from about 60% to about 99% of the liquid nondigestible oil and from about 1% to about 40% of the solid polyglycerol ester particles. Preferably, this mixture will comprise from about 80% to about 99% liquid nondigestible oil and from about 1% to about 20% of the solid polyglycerol ester particles, more preferably from about 85% to about 99% liquid nondigestible oil and from about 1% to about 15% of the solid polyglycerol ester particles, even more preferably from about 90% to about 99% liquid nondigestible oil and from about 1% to about 10% of the solid polyglycerol ester particles, and most preferably from about 95% to about 99% liquid nondigestible oil and from about 1% to about 5% of the solid polyglycerol ester particles. The use of higher levels of liquid nondigestible oil (i.e., lower levels of solid polyglycerol ester particles) may be desirable from the standpoint of reducing the waxiness impression left by the solid components of the nondigestible fat composition. However, higher levels of solid polyglycerol ester

particles (i.e., lower levels of liquid nondigestible oil) may be desirable from the standpoint of controlling or preventing passive oil loss associated with the ingestion of compositions containing such liquid nondigestible oils.

5 The combination of liquid nondigestible oil and solid polyglycerol ester particles is typically formed by simply mixing the liquid and solid components together, by heating the mixture until the solid polyglycerol ester material dissolves in the oil and then by cooling the mixture to a suitable crystallization temperature, e.g., room temperature, which causes polyglycerol ester particles to form.

10 The specific size of the polyglycerol ester particles formed in the fat compositions herein will be dependent upon the rate at which the heated combination of oil and dissolved solid is cooled. As used herein, cooling rate is defined as the temperature differential between (a) the heated oil/dissolved solid combination and (b) the cooled crystallized liquid/solid particle combination, divided by the time taken to create this temperature differential. Generally the greater the cooling rate employed in forming the fat compositions herein, the smaller will be the particles of solid polyglycerol ester material dispersed in such compositions. Desirable cooling rates for use in forming the fat compositions herein are typically greater than 0.6°C/min. (1° F/min.), preferably greater than 2.8°C/min. (5°F/min.), more preferably greater than 5.6°C/min. (10°F/min.), and most preferably greater than 27.8°C/min. (50°F/min.). When the nondigestible fat compositions herein are to be formed in situ, for example, within a food product of which they form a part, then the type and concentration of the fat composition components should be selected so that the cooling profile experienced by the food product will result in formulation of the desired amount and size of the solid polyglycerol ester particles within the food product.

25 The formation of thin nondigestible particles according to the present invention provides especially efficient passive oil loss control for the resulting fat composition. Such efficiency permits a reduction in solids content of the nondigestible fat to relatively low levels (e.g., to from about 1% to about 15%). This reduction in solids levels required for passive oil loss control, together with the minimal/no change in solids between typical room and body temperatures, leads to nondigestible fats having a less waxy tasting impression.

30 Both the liquid nondigestible oil and the solid nondigestible polyglycerol ester

components, as well as their respective concentrations, are selected in order to provide nondigestible fat compositions having certain physical characteristics. In the first place, the nondigestible fats of the present invention should exhibit a relatively flat Solid Fat Content (SFC) profile slope across the temperature range of from typical room temperature to body temperature, i.e. from 70°F to 98.6°F. The SFC profile slope between these temperatures should be from 0 to about -0.75% solids/°F, preferably from 0 to about -0.5 %solids/°F, more preferably from 0 to about -0.3 %solids/°F, and most preferably from 0 to about -0.1 %solids/°F. The method for determining the SFC profile slope of the compositions herein is described hereinafter in the Analytical Methods section.

The nondigestible fat compositions of the present invention should also exhibit particular Thixotropic Area Values. These Thixotropic Area Values are determined by a procedure which reflects the apparent viscosity and thixotropy of the nondigestible fat composition when it is crystallized by cooling according to the cooling profile that will be encountered when the fat is used in any given end use food product. For example, in the case of nondigestible fats of the present invention, this can approximate the cooling profile of a potato chip, and such a cooling profile will be typical of other deep fried salted snack products. Nondigestible fat compositions of the present invention should typically exhibit Thixotropic Area Values of about 10 kilopascals/second (kPa/sec) or greater, preferably about 25 kPa/sec or greater, more preferably about 45 kPa/sec or greater, even more preferably about 70 kPa/sec or greater, most preferably about 80 kPa/sec or greater. Thixotropic Area Values are determined by the method described hereinafter in the Analytical Methods section.

E. Food Products with Nondigestible Fat Compositions

The nondigestible fat compositions of the present invention can be used in various edible fat-containing products including foods, beverages and pharmaceuticals, either alone or in combination with other materials such as digestible fats and oils. In particular, the nondigestible fats of the present invention can be optionally formulated with a digestible triglyceride fat or oil. Generally, these formulations can comprise from about 10% to 100% nondigestible fat and from 0% to about 90% digestible triglyceride fat or oil. Preferably, these formulations comprise from 35% to 100%, more preferably from about 50% to about 100% and most preferably from about 75% to about 100%

nondigestible fat, and from 0% to about 65%, more preferably from 0% to about 50%, and most preferably from 0% to about 25%, digestible triglyceride fat or oil. Because of the potential caloric impact of these triglyceride fats or oils, it is desirable to minimize the level at which they are combined with the nondigestible fats of the present invention.

- 5 As used herein, the term "triglyceride oil" refers to those triglyceride compositions which are fluid or liquid above about 25°C. Although not a requirement, the triglyceride oils useful in the present invention can include those which are fluid or liquid below 25°C. These triglyceride oils consist primarily of triglyceride materials, but can also include residual levels of other components such as mono- and diglycerides.
- 10 To remain fluid or liquid at temperatures below 25°C, the triglyceride oil contains a minimal amount of glycerides having melting points higher than about 25°C so as to limit the solids increase when the triglyceride oil is cooled. It is desirable that the triglyceride oil be chemically stable and resistant to oxidation.

- Suitable triglyceride oils can be derived from naturally occurring liquid
- 15 vegetable oils such as cottonseed oil, soybean oil, safflower oil, corn oil, olive oil, coconut oil, palm kernel oil, peanut oil, rapeseed oil, canola oil (i.e., rapeseed oil low in erucic acid), sesame seed oil, sunflower seed oil, and mixtures thereof. Also suitable are liquid oil fractions obtained from palm oil, lard and tallow by, for example, graining or directed interesterification, followed by separation of the oils. Oils predominating in
- 20 glycerides of unsaturated acids may require partial or touch hydrogenation to maintain flavor, but care should be taken not to greatly increase the amount of glycerides melting above 25°C. When oils are selected which have a larger amount of solids melting between 25° and 40°C than are desirable, it can be necessary to separate out the solids. For example, refined and slightly hydrogenated, and filtered soybean oil is suitable, as
- 25 well as refined cottonseed oil.

- As used herein, the term "triglyceride fat" refers to those triglyceride compositions which are solid or plastic above about 25°C. These solid or plastic fats can be derived from plants or animals or can be edible synthetic fats or oils. For example, animal fats such as lard, tallow, oleo oil, oleo stock, oleo stearin and the like
- 30 which are solid at room temperature can be utilized. Also, triglyceride oils, e.g. unsaturated vegetable oils, can be converted into plastic fats by partial hydrogenation of the unsaturated double bonds of fatty acid constituents of the oil followed by

conventional chilling and crystallization techniques or by proper mixture with sufficient triglycerides which are solid at room temperature to form a rigid interlocking crystalline structure which interferes with the free-flowing properties of the liquid oil. See Purves et al; U.S. Patent 3,355,302; Issued November 28, 1967, and Darragh et al; U.S. Patent 3,867,556; Issued February 18, 1975 (both incorporated herein by reference), for further examples of solid or plastic fats. Because the solid or plastic fats add an appreciable level of solids, their inclusion can cause adverse effects on the organoleptic properties, in particular waxiness, of the edible fat-containing products of the present invention.

Triglyceride fats and oils useful in the nondigestible fats of the present invention can include certain triglycerides in which one, two or three of the OH groups of the glycerol molecule have been substituted with acetyl, propionyl, butyryl, caproyl, caprylyl, or capryl radicals, and the remaining OH groups of the glycerol molecule (if any) have been substituted with acyl radicals of saturated or unsaturated fatty acids having from 12 to 24 carbon atoms.

The nondigestible fat compositions of the present invention can also be used in combination with reduced calorie medium chain and mixed medium/long chain triglycerides such as are disclosed in Ehrman et al; U.S. Pat. 4,888,196; Issued December 19, 1989 and Seiden; European Patent Application 322,027; Published June 28, 1989, both incorporated herein by reference.

The nondigestible fat compositions of the present invention can be used in or as shortening and oil products. These shortening and oil products can be used in frying applications such as preparation of french fried potatoes, potato chips from potato slices or fabricated potato pieces, potato sticks, corn chips, tortilla chips, donuts, chicken, fish, and fried pies (e.g. turnovers). The shortening and oil products can also be used in preparing baked goods in any form, such as mixes, shelf-stable baked goods, and frozen baked goods, including, but not limited to, cakes, granola bars, brownies, muffins, bar cookies, wafers, biscuits, pastries, pies, pie crusts, and cookies, including sandwich cookies, chocolate chip cookies, particularly storage stable dual-texture cookies as disclosed in Hong et al; U.S. Patent 4,455,333; Issued June 19, 1984. These baked goods can contain fruit, cream, or other fillings. Other baked good uses include breads and rolls, crackers, pretzels, pancakes, waffles, ice cream cones and cups, yeast-raised bake goods, pizza and pizza crust, and baked farinaceous snack products and other

baked salted snacks.

Other edible fat-containing products which contain the nondigestible fat compositions of the present invention include ice cream, frozen desserts, cheese, cheese spreads, meats, meat analogs, chocolate confections, salad dressings, mayonnaise, margarine, spreads, sour cream, yogurt, coffee creamer, peanut butter, extruded snacks
5 such as corn curls, corn puffs, pellet snacks, half products and other extruded snacks based on corn or other cereal grains such as wheat, rice and the like, roasted nuts and beverages such as milkshakes.

Edible fat-containing products according to the present invention can include
10 noncaloric or reduced calorie sweeteners alone or in combination with bulking agents. These noncaloric or reduced calorie sweeteners include, but are not limited to, aspartame, saccharin, alitame, thaumatin, dihydrochalcones, acesulfame, and cyclamates.

Bulking or bodying agents which can be useful in edible fat-containing products
15 containing the nondigestible fat compositions herein include partially or wholly nondigestible carbohydrates, for example, polydextrose and cellulose or cellulose derivatives, such as D,L-sugars, carboxymethylcellulose, carboxyethylcellulose, hydroxypropylcellulose, methylcellulose, hydroxypropyl methylcellulose, and microcrystalline cellulose. Other suitable bulking agents include gums (hydrocolloids),
20 starches, dextrans, fermented whey, tofu, maltodextrins, polyols, including sugar alcohols, e.g., sorbitol and mannitol, and carbohydrates, e.g., lactose.

The edible fat-containing products containing the nondigestible fat compositions herein can also include dietary fibers. By "dietary fiber" is meant complex carbohydrates resistant to digestion by mammalian enzymes, such as the carbohydrates
25 found in plant cell walls and seaweed, and those produced by microbial fermentation. Examples of these complex carbohydrates are brans, celluloses, hemicelluloses, pectins, gums and mucilages, seaweed extract, and biosynthetic gums. Sources of the cellulosic fiber include vegetables, fruits, seeds, cereals, and man-made fibers (for example, by bacterial synthesis). Commercial fibers such as purified plant cellulose, or cellulose
30 flour, can also be used. Naturally occurring fibers can be used, such as psyllium and fibers from whole citrus peel, citrus albedo, sugar beets, citrus pulp and vesicle solids, apples, apricots, and watermelon rinds.

These dietary fibers can be in a crude or purified form. The dietary fiber used can be of a single type (e.g., cellulose), a composite dietary fiber (e.g., citrus albedo fiber containing cellulose and pectin), or some combination of fibers (e.g., cellulose and a gum). The fibers can be processed by methods known to the art.

5 The nondigestible fat compositions of the present invention can also be fortified with vitamins and minerals, particularly the fat-soluble vitamins. The fat-soluble vitamins include vitamin A, vitamin D, and vitamin E and their precursors. See Mattson; U.S. Patent 4,034,083; Issued July 5, 1977 (herein incorporated by reference) which discloses fat-soluble vitamins useful in fortifying polyol fatty acid polyester.

10 Various other ingredients typically present in fat products can also be included in the nondigestible fat compositions of the present invention. These other ingredients include stabilizers to help protect against oxidative deterioration at high temperatures. Silicone oils, particularly methyl and ethyl silicone oils, are useful for this purpose. Methyl silicones have also proven effective in reducing the rate of polymerization during
15 frying. Other additives typically included in fat products such as minor amounts of optional flavorings, emulsifiers, anti-spattering agents, anti-sticking agents, antioxidants or the like can also be present.

F. Alternate Utility for the Solid Polyglycerol Ester Particles

It has been found that the solid polyglycerol ester particles useful as passive oil
20 loss control agents in the nondigestible fat compositions herein, are also effective for use as thickening agents in conventional digestible triglyceride oils or oil-containing products. Accordingly, these solid polyol polyester particles can be used as "thickening agents" or "hardstocks" by blending them in amounts of about 1% to about 40% (typically 1% to 15%, more typically 1% to 10%, and most typically 1% to 8%) with
25 liquid digestible oils in the formulation of cooking and salad oils or semi-solid food products such as shortenings, as well as other food products which contain a combination of fat and non-fat ingredients, e.g., margarines, mayonnaise, frozen dairy desserts and the like. The oils for these compositions can comprise conventional digestible triglyceride oils such as cottonseed, corn, canola or soybean, or medium or
30 medium and long chain triglycerides.

G. Analytical Methods

A number of parameters used to characterize elements of the present invention

are to be quantified by experimental analytical procedures. Each of these procedures is described in detail as follows:

1. Fatty Acid Composition of Polyol Polyesters

5 The fatty acid composition (FAC) of the polyol polyesters is determined by gas chromatography, using a Hewlett-Packard Model S712A gas chromatograph equipped with a flame ionization detector and a Hewlett-Packard Model 7671A automatic sampler. The chromatographic method used is described in Official Methods and Recommended Practices of the American Oil Chemists Society, 4th Ed., 1989, Procedure 1-Ce62 (incorporated herein by reference).

10 2. Ester Distribution of Sucrose Polyesters

The relative distribution of the individual octa-, hepta-, hexa- and penta- esters, as well as collectively the tetra- through mono- esters, of the sucrose polyesters can be determined using normal-phase high performance liquid chromatography (HPLC). A silica gel-packed column is used in this method to separate the polyester sample into the
15 respective ester groupings noted above. Hexane and methyl-t-butyl ether are used as the mobile phase solvents. The ester groupings are quantitated using a mass detector (i.e. an evaporative light-scattering detector). The detector response is measured and then normalized to 100%. The individual ester groups are expressed as a relative percentage.

20 3. Slope of Solid Fat Content (SFC) Profile of Nondigestible Fat Measured in °F

Before determining the SFC values, a sample of the nondigestible fat is heated to a temperature of 140°F (60°C) or higher for at least 30 minutes or until the sample is completely melted. The melted sample is then tempered as follows: at 80°F (26.7°C) for 15 minutes; at 32°F (0°C) for 15 minutes; at 80°F (26.7°C) for 30 minutes; at 32°F
25 (0°C) for 15 minutes. After tempering, the SFC values of the sample at temperatures of 50°F (10°C), 70°F (21.1°C), 80°F (26.7°C), 92°F (33.3°C), and 98.6°F (37°C) are determined by pulsed nuclear magnetic resonance (PNMR) after equilibration for 30 minutes at each temperature. The slope of the SFC profile in % solids/°F is calculated by subtracting the SFC value at 70°F (21.1°C) from the SFC value at 98.6°F (37°C)
30 and then dividing by 28.6. The method for determining SFC values by PNMR is described in J. Amer. Oil Chem. Soc., Vol. 55 (1978), pp. 328-31 (herein incorporated by reference, and A.O.C.S. Official Method Cd. 16-81, Official Methods and

Recommended Practices of The American Oil Chemists Society, 4th. Ed., 1989, (herein incorporated by reference).

4. Complete Melting Point of Polyol Polyesters by Differential Scanning Calorimetry (DSC)

5 The complete melting point of the polyol polyester material or polyol polyester-containing particles used in this invention can be determined by DSC as follows:

Equipment:

Perkin-Elmer 7 Series Thermal Analysis System, Model DSC7, manufactured by Perkin-Elmer, Norwalk, CT.

10 Procedure:

1. Sample of polyol polyester material or polyol polyester-containing blend is heated to at least 10°C above the temperature at which all visible solids are melted and mixed thoroughly.

2. 10 ± 2 mg of sample is weighed into sample pan.

15 3. A scan is performed from about 10°C above the temperature at which all visible solids are melted to -60°C at 5°C per minute.

4. The temperature of the sample is maintained at -60°C for 3 minutes and scanned from -60°C to the original starting temperature at 5°C per minute (i.e., to about 10°C above the temperature at which all visible solids are melted).

20 5. The complete melt point is the temperature at the intersection of the base line (i.e. specific heat line) with the line tangent to the trailing edge of the last (e.g., highest temperature) endothermic peak.

5. Thixotropic Area Value

25 The nondigestible fat compositions of the present invention exhibit certain rheological characteristics (i.e. apparent viscosity and thixotropy) which correlate to the extent of passive oil loss control that such compositions provide. The method described herein may be used to determine the Thixotropic Area Value of a fat composition wherein the solid component has crystallized via the cooling profile of the end-use product to form a three-dimensional, solid-like structure.

30 Thixotropic Area can be expressed in terms of the dimensions of energy per unit volume of sample being sheared, which is an indication that energy is required to break down the three-dimensional, solid-like structure of the material. (See Schram, G.

Introduction to Practical Viscometry, (1981), pp. 17-19, Gebruder Haake, West Germany.) Thus, Thixotropic Area may be considered a relative measurement of the three-dimensional, solid-like network of the fat composition that exists prior to shearing. In this method, shear stress is measured as a function of shear rate between $0s^{-1}$ and $800s^{-1}$ using a cone and plate rheometer. The shear rate is first increased for 7.5 minutes and then decreased for 7.5 minutes at $37.8^{\circ}C$. The Thixotropic Area is the area of the hysteresis between the ascending and descending flow curves. Nondigestible fat compositions which have Thixotropic Area Values of at least about 10 kPa/sec will exhibit passive oil loss control when ingested.

10 a). Calibration of Recorder

A cooling profile template (see Figure 1) is placed on an X-Y recorder (Houston Instruments Model 200) so that time is the X-axis. The cooling profile used should approximate that of the end-use product. In this case, the cooling profile used is an approximation of the cooling profile of a potato chip and is typical of all deep fat fried salted snack products. The recorder switches are set to the parameters described on the

15 template and then calibrated in the following manner:

1. Calibrator set to 50 mv.
2. ZERO potentiometer adjusted until pen indicates $50^{\circ}F$ on recorder.
3. Calibrator is set to 190 mv.
- 20 4. SPAN potentiometer is adjusted until pen indicates $190^{\circ}F$ on recorder.

The above steps 1-4 are repeated until the pen indicates the proper temperature without adjustment. The strip chart recorder is then attached to the analog output of a thermocouple reader (Omega #199A).

 b). Sample Preparation

25 A nondigestible fat sample is heated above $180^{\circ}F$ until completely melted and then thoroughly mixed. Eight (8) grams of the sample are then weighed into an aluminum weighing pan (VWR Scientific #25433-008). A thermocouple (Omega #5TC-T-36-36 0.005 inch type T) is submerged in the sample in approximately the center of the pan, care being taken to keep the thermocouple tip from touching the

30 bottom of the pan. The pan is then placed on a hot plate and heated to approximately $240^{\circ}F$. (240° is the estimated surface temperature of a potato chip after it is removed from the fryer). This temperature may have to be adjusted to approximate the cooling

profile of the particular end-use product into which the fat composition being tested will eventually be incorporated. When the appropriate temperature is reached, the recorder is started and the pan is removed from the hot plate and placed on top of a lab bench. The temperature of the sample is controlled so as to approximately track ($\pm 5^\circ$) the cooling curve shown on the template. This is achieved by providing gentle agitation to the pan to accelerate cooling and removing of the pan from the lab bench top to slow the cooling rate. This cooling process takes approximately 3 minutes to complete, after which time the thermocouple is removed. The nondigestible fat sample is then tempered for at least 30 minutes at a temperature which is typical of the storage temperature generally encountered by the end-use product into which the nondigestible fat sample will eventually be incorporated (e.g. 70°F for a potato chip) prior to measurement of the thixotropic area.

c). Rheometer Setup

The rheometer (Contraves Rheomat 115A with 2, 5 and 7 cm cones; 2° angle) is interfaced with a computer and set up under the following conditions:

Program Setup

Sensitivity	1.0	
First minimum shear rate (s-l)	0.000	
Time at minimum shear rate (s)	120.0	Hold time to allow sample temperature equilibration
Ascending ramp time (s)	450.0	7.5 minute scan 0 to 800 s-l
Maximum shear rate (s-l)	800.000	
Hold time (s)	1.0	
Second minimum shear rate (s-l)	0.000	
Descending ramp time (s)	450.0	7.5 minute scan 800 to 0s^{-1}

Data Output Conditions

Printout of measured points 1 to 15
 Calculate Thixotropic Area
 Printout results

d) Cone Selection

Using a CP-8 (2 cm) cone, measure the Thixotropic Area of the sample according to this method. If the Thixotropic Area Value is greater than 200 kPa/s, maximum accuracy has been attained. If the Thixotropic Area Value is between 50 and 200 kPa/s, the method should be repeated using a CP-6 (5 cm) cone. If the Thixotropic Area Value is between 0 and 50 kPa/s, the method should be repeated using the CP-10 (7 cm) cone.

e) Torque Calibration

The rheometer is calibrated for torque by lifting the measuring head away from the plate and then adjusting the torque calibration knob on the control panel of the rheometer until the torque meter to the left of the adjustment knob reads "+000" with the "+" flashing.

f) Temperature Calibration

The temperature of the sample during analysis should be maintained at $37.8 \pm 0.1^\circ\text{C}$. After setting the recirculating bath to achieve approximately 37.8°C , the plate temperature is checked by applying a small amount of oil to the plate, positioning the cone onto the plate, inserting the thermocouple probe into the gap between the cone and the plate, and then allowing a few minutes for the temperature to equilibrate. The temperature is then read with the bath temperature being adjusted until the plate temperature is $37.8 \pm 0.1^\circ\text{C}$.

g) Sample Analysis

Approximately 4 grams of the tempered nondigestible fat sample is applied to the rheometer plate. The cone assembly is then lowered slowly onto the sample and seated firmly on the plate. At this point, the flow curve program is initiated. Upon completion of the run, a report is printed out listing the first 15 data points on the flow curve and the calculated thixotropic area. The Thixotropic Area is the hysteresis area between the ascending and descending flow curves and is reported as the Thixotropic Area Value (Kpa/sec.).

6. Average Degree of Glycerol Polymerization

The "average degree of glycerol polymerization" (\bar{n}) is a molar quantity which describes the the average number of glycerol moieties in the polyglycerol ester species comprising a polyglycerol ester mixture. The average degree of glycerol

polymerization is calculated from an experimentally determined distribution of the weight percentages of the individual polyglycerol ester species which make up a given mixture of polyglycerol esters.

The distribution of polyglycerol ester species in a polyglycerol ester sample can be determined as follows: the polyglycerol ester sample is transesterified with sodium methoxide in refluxing methanol. The sodium methoxide is removed from the resulting solution by treatment with an anion exchange resin. The methanolic solution of polyglycerols and resulting methyl esters is extracted with hexane to remove the methyl esters. Finally, the methanol is evaporated, leaving the mixture of unesterified polyglycerols. The polyglycerols thus obtained are derivatized with a 5/1 (by volume) mixture of trimethylsilyl-imadazole and bis(trimethylsilyl)trifluoroacetamide in pyridine to form trimethylsilyl ethers. The sample is analyzed by GC using a short (18 inches by 1/8 inch ID), packed column (3% JXR on 100/120 mesh Gas Chrom Q), on column injection and flame ionization detection. The GC method is essentially that used for the separation of intact mixtures of mono-, di-, and triglycerides described in JAOCS, 58, (1981) pages 215-227.

The average degree of glycerol polymerization (\bar{n}) can then be calculated from the determined distribution of polyglycerol species in the sample according to the following equation:

$$\bar{n} = \frac{\sum_{n=1}^{\infty} n \times \frac{\text{Wt \% } G_n}{\text{MW}_{G_n}}}{\sum_{n=1}^{\infty} \frac{\text{Wt \% } G_n}{\text{MW}_{G_n}}}$$

where $\text{Wt \% } G_n$ = weight % in the sample of a polyglycerol species having n repeating units

MW_{G_n} = the molecular weight of a polyglycerol ester species having n repeating units = $n(74) + 18$

7. % Esterification of Polyglycerol Ester Mixture

The % esterification of a polyglycerol ester sample is the average degree of polyglycerol esterification expressed on a mole percent basis. The % esterification is calculated indirectly from the Saponification Value, the Acid Value and the average degree of glycerol polymerization of a polyglycerol ester sample. The analytical methods for determining the Saponification Value and the Acid Value of a polyglycerol

ester sample are as follows:

Saponification Value

The solid polyglycerol ester sample can be saponified with refluxing alcoholic KOH according to the procedure described in Official Methods and Recommended Practices of the American Oil Chemists Society, 4th Ed., 1989, Procedure Cd 3-25. The resulting fatty acid soaps are titrated with standardized HCl to a phenolphthalein endpoint. A blank (no sample added) is also run through the procedure and titrated.

The Saponification Value can then be calculated according to the following equation:

10
$$SV = ((B - S) \times N \times 56.1) / W$$

Where B = volume (mls) HCl required to titrate the blank

S = volume (mls) HCl required to titrate the sample

N = normality of the HCl

W = sample weight in grams

15 Acid Value

The solid polyglycerol ester sample can be titrated with standardized KOH to a phenolphthalein endpoint. The procedure is described in Official Methods and Recommended practices of the American Oil Chemists Society, 4th Ed., 1989, Procedure Cd 3a-63. A blank (no sample added) is titrated also.

20 The Acid Value can then be calculated according to the following equation:

$$AV = ((A - B) \times N \times 56.1) / W$$

where A = volume in mls of KOH required to titrate the sample

B = volume in mls of the KOH required to titrate the blank

N = normality of the KOH

25 W = sample weight in grams

From the Saponification Value and the Acid Value, the "Ester Value" (EV) of the polyglycerol ester sample can then be calculated. The Ester Value of a given polyglycerol ester sample is the difference between the saponification value (SV) and the acid value (AV) of the sample.

30 From the Ester Value, a Corrected Ester Value can then be calculated. The "Corrected Ester Value" (EV_{cor}) of a given polyglycerol ester sample is the calculated

ester value of a pure sample containing only the polyglycerol esters (i.e., containing no free fatty acid). Corrected Ester Value is calculated according to the following equation:

$$EV_{cor} = \frac{EV}{1 - \frac{\%ffa}{100}}$$

where $\%ffa = AV(0.503)$

Next, an average degree of esterification (i -bar) is calculated from the corrected ester value and the average molecular weight of the polyglycerol (MW_{Gn-bar}). The average degree of esterification (i -bar) is a molar quantity which describes the average number of the hydroxyl groups of the polyglycerol ester sample which are esterified with fatty acids. Thus,

$$i - bar = \frac{(EV_{cor})(MW_{Gn-bar})}{56,100 - (EV_{cor})(avg. MW_{fa} - 18)}$$

where $MW_{Gn-bar} = n-bar(74) + 18$

avg. MW_{fa} = the average molecular weight of the fatty acid ester groups (fa) present in the polyglycerol ester sample calculated from the weight percent fatty acids of the various species as measured by the GCFAC method hereinbefore described according to the equation:

$$avg. MW_{fa} = \sum \frac{wt\%_{fa}}{100} MW_{fa}$$

Lastly, the % esterification is calculated according to the following equation:

$$\% \text{ esterification} = \frac{(i - bar) 100}{n - bar + 2}$$

8. Digestibility of Fat Compositions (Lipase Test)

About 0.5g of the nondigestible fat composition is melted and added to 25ml of a Tris buffer solution (58.4g NaCl (1.0M), 5.3g $CaCl_2 \times 2H_2O$ (36mM), 67.7g Trizma (0.5M) diluted to 1 liter with deionized water and adjusted to pH 8.0 with

concentrated HCl) in a 125ml Erlenmeyer flask. To this mixture is added 1.0ml of a 1.0% sodium taurocholate solution (Sigma Chemical) and 0.5ml of a 45.0% $\text{CaCl}_2 \times 2\text{H}_2\text{O}$ solution. About 5 glass beads are added, the flask stoppered and placed in water bath controlled at 37°C equipped with wrist action shaker. The sample is shaken for 1 hour then 1ml of lipase solution (750mg of lipase (Sigma Chemical type II, crude from porcine pancreas) diluted to 50ml with the Tris buffer solution described above) is added. The mixture is shaken at 37°C for one hour.

The reaction is terminated by the addition of 10ml of concentrated HCl, 25ml of deionized water and 10ml of ethanol. The liberated free fatty acids are extracted with 3-100ml portions of diethyl ether/petroleum ether (1:1 by volume). The combined extracts are washed with 3-100ml portions of deionized water. The organic layer is dried with anhydrous sodium sulfate and filtered through Whatman #41 filter paper. The ether solvents are removed by rotary evaporation at 55°C.

The residue is washed into a 150ml beaker with 2-30ml portions of hot isopropyl alcohol/water (85/15 v/v). The mixture is titrated with standardized 0.1N NaOH solution to a phenolphthalein endpoint. A blank (no added test material) is run through the entire procedure and titrated also. The "lipase ester value" (LEV) is calculated from the following equation:

$$\text{LEV} = (((s-B) \times N \times 56.1)/W) - \text{AV}$$

where S = volume in mls of NaOH required to titrate the sample

B = volume in mls of NaOH required to titrate the blank

N = normality of the NaOH

W = sample weight on grams

AV = acid value of the sample (described hereinabove)

The percent hydrolysis is calculated from the following equation:

$$\% \text{ Hydrolysis} = (\text{LEV}) \times 100 / (\text{EV})$$

where LEV = lipase ester value (hereinabove)

EV = ester value (described hereinabove)

Using these conditions and this enzyme preparation, only the esters of primary hydroxyl groups are hydrolyzed. For example, the LEV of pure triolian (triglyceride containing three oleic acid esters) is 126.9, the SV is 190.4 and the % hydrolysis is 66.7%. However, the triolian also contains hydroxyl groups other than primary

hydroxyl groups which must be accounted for in determining digestibility of the triolian even though these other hydroxyl groups are not hydrolyzed under the conditions of this method. Therefore, it is assumed that triolian, a conventional triglyceride, is 100% digestible and the value of 66.7% hydrolysis obtained for the triolian sample according to this method is normalized to 100%. According to the definition of nondigestibility set forth hereinbefore in the Definitions section, only about 70% of the sample can be hydrolyzed versus a triglyceride by the enzymes in this lipase test. Therefore, for a fat composition to be considered nondigestible, the % hydrolysis value obtained for the sample according to this method should be 46.7% or less, preferably 13.3% or less, more preferably 6.7% or less.

9. Thickness of Polyglycerol Ester Particles (Light Microscopy)

The thickness of the solid polyol polyester particles formed in the nondigestible fat composition herein may be estimated at room temperature with a Nikon Microphot video-enhanced light microscope (VELM) using Hoffman Modulation Contrast (HMC) optics according to the following method:

1. A small portion (i.e., 1-10mg) of the nondigestible fat sample with the solid polyglycerol ester particles dispersed therein is placed on a microscope slide and covered. The slide is placed under the microscope.
2. The sample is examined using a HMC 100X oil objective as the standard lens in conjunction with a 10X eyepiece lens.
3. A microscope-mounted video camera and associated controller are used for video enhancement to facilitate differentiation between the sample and the background.
4. The thickness of the solid polyol polyester particles is measured in μm .

This method permits differentiation of particles having thicknesses just within the resolution of the VELM (approximately 0.2 - 0.5 μm). Particle thickness of particles having smaller dimensions can be determined by the Freeze Fracture Method described hereinafter.

(Note: No special sample preparation is required, other than obtaining a representative sample. The samples should be melted and cooled ambiently.)

Reference: Robert Hoffman, "The Modulation Contrast Microscope: Principles and Performances", Journal of Microscopy, Vol. 110, Pt 3, August 1977, pp. 205-222.

10. Thickness of Solid Polyol Polyester Particles-Freeze Fracture Transmission

Electron Microscopy

The three-dimensional topography of particles of polyol polyesters and their size can be determined by a freeze-fracture transmission electron microscopy (ff-tem) method.

5 This freeze-fracture method is carried out as follows:

1. The outside cavity of a freezing container is filled with liquid N₂ and the inner dewar of the freezing container is filled with liquid ethane (normal melting temperature of -172°C). The ethane is allowed to freeze.

2. A small amount (1-2 ul) of the nondigestible fat sample with the solid
10 polyol polyester particles dispersed therein is placed in the well of a gold-plated Balzers specimen holder. (Note: for very fluid samples, 1-2 ul of sample is placed on a gold planchet (Balzers) and another planchet is placed on top of the first to form a sandwich.)

3. Most of the frozen ethane in the dewar is melted by inserting a metal heat sink (e.g., tweezers) into the dewar.

15 4. Immediately after melting the ethane, the specimen holder containing the nondigestible fat sample is picked up using a pair of tweezers and rapidly plunged into the liquid ethane.

5. After a few seconds, the specimen holder is removed from the ethane, quickly touched to the tip of a camel's hair brush to remove excess ethane, and
20 immediately immersed in the liquid N₂ to keep the sample cold.

6. The sample is transferred under liquid N₂ to a JEOL JFD-9000C sample holder and then transferred into the chamber of a JEOL JFD-9000C freeze-fracture unit. The temperature of the unit should be about -175°C. Vacuum should be at least 8×10^{-7} torr.

25 7. A knife is cooled to a temperature of about -165°C.

8. The sample is fractured in the JEOL chamber using the pre-cooled knife.

9. Platinum-carbon is deposited onto the fractured sample at a 45° angle for 4.5 seconds, followed by carbon deposition at a 90° angle for 25 seconds to form a
30 replica of the fractured sample. The high voltage is 2500 and the current is 70 mA.

10. The samples are removed from the freeze fracture unit and cleaned using 3 washes of chloroform.

11. The replica is picked up on a 300 mesh copper EM grid and examined in a transmission electron microscope.

12. Images are recorded on negative film and positive prints are made from the negatives.

5 13. The thickness of the polyol polyester particles is measured in nm.

References:

Rash, J.E. and Hudson, C.S., Freeze Fracture: Methods, Artifacts, and Interpretations, New Haven Press, New York, 1979.

10 Stolinski and Breathnach, Freeze Fracture Replication of Biological Tissues, Academic Press, London, 1975.

Steinbrecht and Zierold, Cryotechniques in Biological Electron Microscopy, Springer-Verlag, Berlin, 1987.

H. Specific Examples

15 Specific preparation of the nondigestible fat compositions of the present invention is illustrated by the following examples:

Example I

Solid Polyglycerol Ester Preparation

20 200 grams of a wide distribution polyglycerol containing di- through heptaglycerols (average $\bar{n} = 3.35$) is prepared in an industrial scale process using the procedure described in Babayan; U.S. Patent 3,637,774; Issued January 25, 1972 (incorporated herein by reference). The polyglycerol is then fractionated on a laboratory scale Pope wiped film evaporator to remove water, glycerine, diglycerol, and triglycerol. The fractionated polyglycerol is run through the evaporator at a rate of 0.8 to 1.2 g/min., at 190-191°C and a pressure of 0.15 to 0.8 mm HG. About 74g of distillate and about 106g of product are isolated (most of the water flashes off and is lost to the vacuum system). The final polyglycerol product contains only trace amounts of water and glycerine, and has reduced diglycerol and triglycerol levels, with an \bar{n} of 4.76. The average degree of polymerization is 4.76 and the average molecular weight is 370.2.

30 The reaction is conducted in a 100 ml spherical glass reaction vessel equipped with a nitrogen inlet and magnetic stirrer. The temperature is controlled by means of an oil bath, thermometer and temperature controller. Approximately 5.00 grams (13.5

mMoles) of the finished polyglycerol described above are added to the reactor along with 150 ml of dry pyridine and 75 ml of dry dimethylformamide. The polyglycerol is allowed to dissolve at room temperature, and then a mixture of palmitoyl chloride (3.30g, 12.0 mMoles) and stearoyl chloride (24.2g, 80.0 mMoles) is added. The clear, slightly yellow reaction mixture is heated to about 50-52°C for approximately 4.0 hours with stirring under an atmosphere of dry nitrogen.

Next, most of the pyridine and dimethylformamide are removed from the reaction mixture by rotary evaporation at 70-80°C and a pressure of about 15 mm HG. The crude product is dissolved in 200 ml of dichloromethane and transferred to a 500 ml separatory funnel. The solution is washed with 2-200 ml portions of 10% aqueous HCL and finally 2-200 ml portions of distilled deionized water. The organic phase is dried with anhydrous sodium sulfate, filtered and the solvent removed by rotary evaporation.

The resulting solid polyglycerol ester has the following attributes:

Saponification Value:	179.8
Acid Value:	32.0
Corrected Ester Value:	176.2
Melting Point:	54.8
n-bar:	4.97
i-bar:	6.66
% esterification:	95.6
Avg. MW _{FA} :	278.5
Fatty Acid Content:	
C ₁₄	0.1
C ₁₆	11.4
C ₁₇	0.2
C _{18:0}	86.3
C _{18:1}	0.6
C ₂₀	0.6

15 Fat Composition Preparation

Four (4) grams of this solid polyglycerol ester product and 96 grams of a liquid sucrose polyester, in which the sucrose is substantially completely esterified with fatty

acid groups of cottonseed oil, are mixed and heated until all the solids are dissolved. The mixture is then allowed to cool back to room temperature at a rate of 33.3oF/min. The resulting fat composition is suitable for use as a food fat. Because the fat composition has a Thixotropic Area Value of 19.0, it does not produce passive oil loss which would otherwise result if only liquid sucrose polyester were used. Also, since the level of solids in these fat compositions is so low, food products containing these fat compositions will not be waxy tasting.

The above liquid sucrose polyester has the attributes set forth in Table I.

10

Table I

<u>FATTY ACID COMPOSITION</u>	<u>LIQUID SUCROSE POLYESTER %</u>
C ₁₄	0.2
C ₁₆	17.0
C ₁₇	0.1
C _{18:0}	5.3
C _{18:1}	36.8
C _{18:2}	38.4
C _{18:3}	0.4
C ₂₀	---
<u>ESTER DISTRIBUTION</u>	
% Octa	78.8
% Hepta	20.8
% Hexa	<0.1
% Lower	0.3

Example IISolid Polyglycerol Ester PreparationPolyglycerol Preparation

15

A polyglycerol is prepared in an industrial scale process using the procedure described by Babayan; U.S. Patent 3,637,774; Issued January 25, 1972. The

polyglycerol is fractionated to remove water, glycerine and some of the diglycerol. The resulting narrow distribution polyglycerol had an n-bar of 3.16 and contains mostly di-, tri-, and tetraglycerol with small amounts of penta- through hepta-glycerol. The average degree of polymerization is 3.16 and the average molecular weight is 251.8.

5 Behenic Methyl Ester Preparation

Behenic methyl esters are made from behenic mono- and diglycerides. 3,950 lbs. of behenic glycerides (monoglycerides 27.5%, diglycerides 67.5%, and Triglycerides 5.0%), 660 lbs. of methanol, and 70 lbs. of sodium methylate solution (25% on methanol) are added to a 750 gallon reactor. The mixture is reacted at 65°C for approximately 2 hours while refluxing the methanol. The agitation is stopped, and the glycerin is allowed to settle for about 2 hours. The glycerin settles to the bottom and is removed through the bottom outlet. An additional 60 lbs. of methanol and 11 lbs. of sodium methylate solution (25% in methanol) are added to the reactor, and the mixture is reacted at about 65°C for one hour. The agitation is stopped, the glycerin is settled for two hours, and removed through the bottom outlet. 1,500 lbs. of water is added to the mixture, stirred for 10 minutes, and settled for one hour. The water is then removed through the bottom outlet of the reactor. The methyl ester is then dried under a vacuum of 5-10 mm Hg at a temperature of 65°C. The methyl ester is flash distilled from the reactor through a condenser and into a receiver. Distillation conditions are 300°F - 440°F and 1-5 mm Hg. The distillate purity is 95% C₂₂, 2% C₂₄, and 2.2% C₂₀. Monoglycerides and glycerine are undetectable in the distillate.

15 Polyglycerol Esterification

The esterification reaction is conducted in a 1 liter spherical glass reaction vessel equipped with a nitrogen inlet, vacuum outlet and a mechanical stirrer. The temperature is controlled by means of a heating mantle, thermometer and temperature controller. 27.4 grams (0.109 Moles) of the polyglycerol described above is added to reactor, along with 263.0g (0.743 Moles) methyl behenate and 1.4 grams (0.010 Moles) potassium carbonate. The pressure in the system is reduced to about 6.0 mm HG while simultaneously raising the temperature to 135°C. The reaction mixture is heated with stirring at this temperature for a total of eleven hours during which time the pressure drops to 0.4 mm HG.

30 The product is refined by slurring the crude reaction mixture with 1% silica gel

followed by filtration to remove the solids. The excess methyl esters are removed on a Pope wiped film evaporator operated at a temperature of 210°C and a pressure of 0.05 mm HG.

The resulting solid polyglycerol ester has the following attributes:

5

Saponification Value:	145.7
Acid Value:	0.1
Corrected Ester Value:	145.7
n-bar:	3.11
i-bar:	3.87
Melting Point:	72.2°C
Degree of Esterification	75.7%
Avg. MW _{fa}	338.9
Fatty Acid Composition:	
C ₁₆	0.2
C _{18:0}	0.5
C _{18:1}	0.1
C _{18:2}	0.1
C ₂₀	2.1
C _{22:0}	94.7
C _{22:1}	0.2
C ₂₄	2.0

Fat Composition Preparation

Four (4) grams of this solid polyglycerol ester product and 94 grams of the liquid sucrose polyester described in Example I are mixed and heated until all the solids dissolve. The mixture is then allowed to cool back to room temperature at a rate of 33.3 °F/min. The resulting fat composition has a Thixotropic Area Value of 44.3, and thus, does not produce passive oil loss when used as a food fat. Also, since the level of solids in this fat compositions is so low, food products containing this fat composition will not be waxy tasting.

15

Example III

Solid Polyglycerol Ester Preparation

A commercially available narrow distribution (n-bar 3.19) PGE (Triodan 55, Lot #00202, Grinsted Denmark) is fractionated to remove most of the monoesters and some of the diesters leaving mostly di-, tri- and tetraesters with small amounts of penta-
5 through heptaester. The starting polyglycerol ester has an i-bar of 1.30 and a degree of esterification of 25%.

The resulting solid polyglycerol ester has the following attributes:

Saponification Value:	159.4
Acid Value:	0.6
Corrected Ester:	159.3
n-bar:	3.54
i-bar:	2.83
Melting Point:	56.2°C
Degree of Esterification:	51.1%
MW _{fa} :	271.2
LEV	1.8
Fatty Acid Composition:	
C ₁₂	0.1
C ₁₄	1.2
C ₁₅	0.1
C _{16:0}	41.2
C _{16:1}	0.2
C ₁₇	0.3
C _{18:0}	55.6
C _{18:1}	0.2
C _{18:2}	0.2
C ₂₀	0.7

10 Fat Composition Preparation

Six (6) grams of this solid polyglycerol ester product and 94 grams of liquid sucrose polyester described in Example I are mixed and heated until all the solids are

dissolved. The mixture is then allowed to cool back to room temperature at a rate of 33.3°F/minute. The cooling brings about crystallization of the solid polyglycerol ester material in the form of small, two-dimensional particles which are dispersed in the liquid nondigestible oil. Figure 2 is a photomicrograph depicting the two-dimensional structure of the solid polyglycerol ester particles. These particles have a thickness of less than about 100 nm as measured by Freeze Fracture Transmission Electron Microscopy described hereinafter in the Analytical Methods section.

The fat composition comprising the solid particles of polyglycerol ester dispersed in the liquid sucrose polyester has a Thixotropic Area Value of 38.0, and thus, does not produce passive oil loss which would otherwise result if only the liquid nondigestible oil were to be used as a food fat. The SFC profile slope of the nondigestible fat composition is -0.1 %solids/°F. As a result of this relatively flat SFC profile slope and the low solids level, food products containing this fat composition will not be waxy tasting.

15

Example IV

Norchip potatoes are used which have been sliced to a thickness of about 0.052 inches (0.13cm). The sliced potatoes are fried in a 5 pound batch fryer at a temperature of 365°F (180°C) for 3 minutes. Approximately 225 potato chips are fried in each of the fat compositions of Examples I, II and III.

20

Ingestion of these potato chips which contain the nondigestible fat compositions will not result in passive oil loss, and the potato chips are not unacceptably waxy tasting.

WHAT IS CLAIMED IS:

1. A nondigestible fat composition useful as a replacement for triglyceride fats or oils in foods, which composition has a Solid Fat Content profile slope between 21.1°C and 37°C of from 0 to -1.26% solids/°C and which composition comprises:
 - A. a liquid nondigestible oil having a complete melting point below 37°C; and
 - B. nondigestible solid particles of a polyglycerol ester dispersed in said oil in an amount sufficient to control passive oil loss upon ingestion of said composition, said nondigestible solid particles having a complete melting point above 37°C, wherein the ester groups forming said polyglycerol ester are formed from long chain fatty acid radicals with at least 40%, preferably at least 50%, more preferably at least 75%, of said long chain fatty acids being saturated and having at least 18 carbon atoms, and wherein said nondigestible particles serve to impart to said composition a Thixotropic Area Value of 10 kPa/sec or greater, preferably 25 kPa/sec or greater, more preferably 45 kPa/sec or greater.
2. The nondigestible fat composition of Claim 1 which comprises from 60% to 99%, preferably from 85% to 99%, of a liquid nondigestible oil, preferably a liquid sucrose fatty acid polyester, and from 1% to 40%, preferably from 1% to 15% of solid polyglycerol ester particles.
3. The nondigestible fat composition of Claim 2 wherein the solid polyglycerol ester contains at least 2, preferably from 3 to 10, glycerol moieties and wherein at least 30%, preferably at least 50% of the hydroxyl groups of the solid polyglycerol ester are esterified.
4. The nondigestible fat composition of Claim 3 wherein in the polyglycerol ester the average degree of glycerine polymerization is from 2 to 10, preferably from 3 to 6.
5. The nondigestible fat composition of Claim 4 wherein the ester groups forming the solid polyglycerol ester comprise (i) ester groups

formed from C₂₂ radicals, (ii) ester groups formed from a mixture of C₁₆ and C₁₈ radicals in a molar ratio of C₁₆:C₁₈ ranging from 50:50 to 0.1:99.9, or (iii) a combination thereof.

6. A food product comprising an edible substrate, preferably a potato chip and from 10% to 100% of the nondigestible fat composition of Claim 1.

7. A thickened digestible oil product comprising:
A. from 85% to 99% of a digestible edible oil; and
B. from 1% to 15% nondigestible solid particles of a solid polyglycerol ester, said nondigestible solid particles having a complete melting point above 37°C, wherein the ester groups forming said polyglycerol ester are formed from long chain fatty acid radicals with at least 40% of said long chain fatty acids being saturated and having at least 18 carbon atoms, and wherein said nondigestible particles serve to impart to said composition a Thixotropic Area Value of 10 kPa/sec or greater.

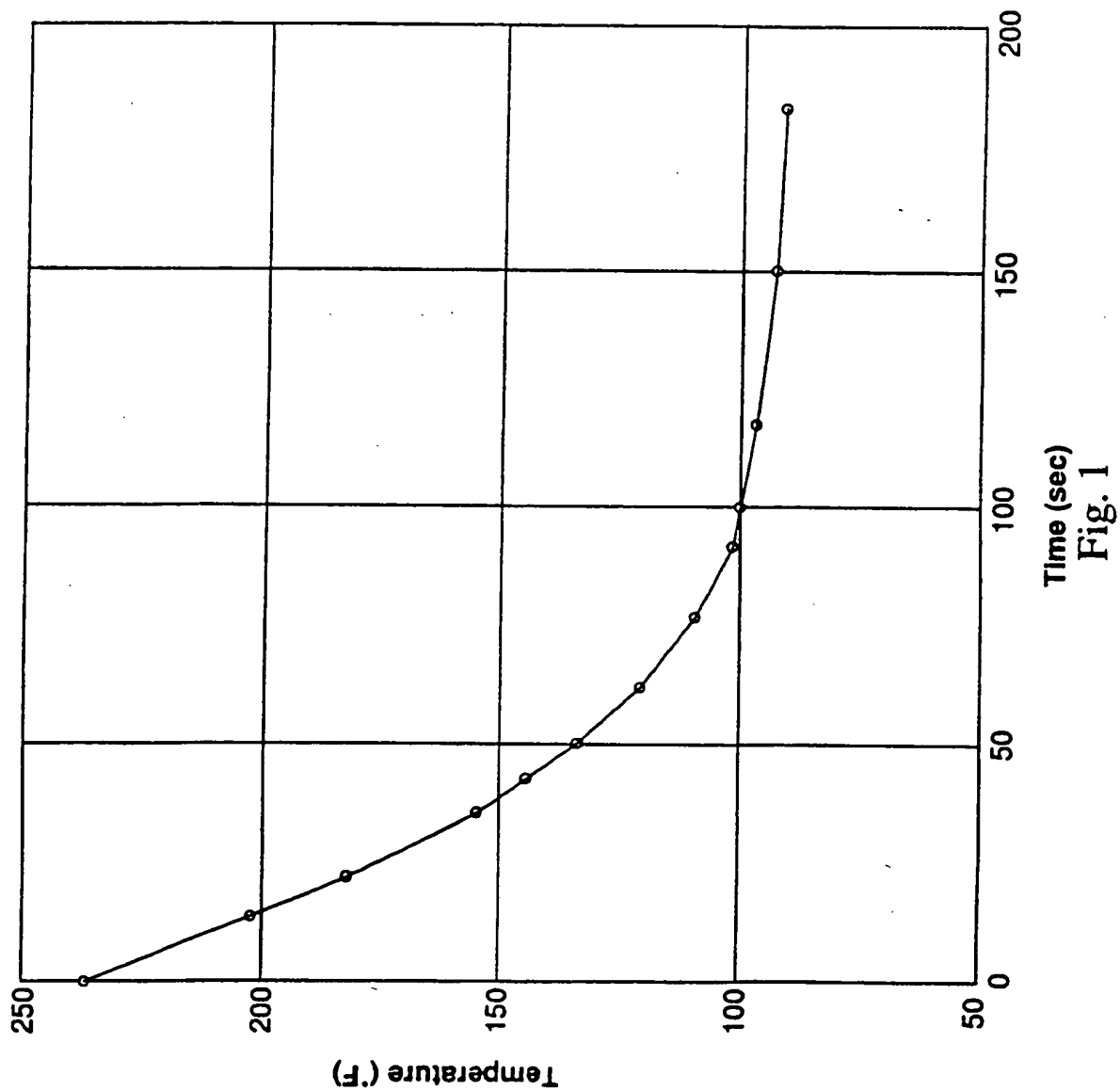


Fig. 1

SUBSTITUTE SHEET

2 / 2

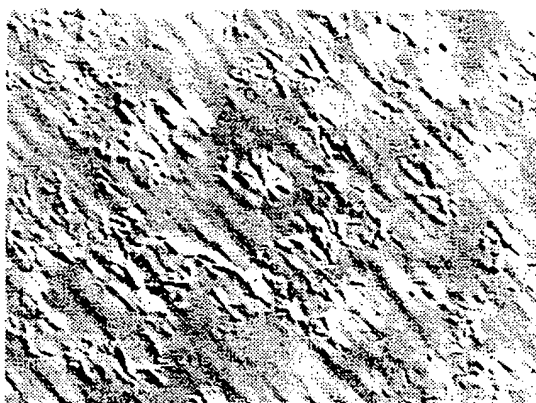


Fig. 2

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 93/10110

A. CLASSIFICATION OF SUBJECT MATTER
IPC 5 A23D9/00 A23L1/308 A23L1/217

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 5 A23D A23L C11C

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	EP,A,0 375 027 (THE PROCTER & GAMBLE COMPANY) 27 June 1990 see page 2, line 36 - page 3, line 28 see page 4, line 40 - line 58 see page 6, line 10 - line 27 see page 7, line 35 - line 44 see page 9, line 22 - line 26 see claims 1,3,5,7-10 ---	1-7
Y	WO,A,91 10368 (THE DOW CHEMICAL COMPANY) 25 July 1991 see page 1, line 1 - page 8, line 2 see page 9, line 33 - page 10, line 22 see example 16 see claims 4,11 --- -/--	1-7

☒ Further documents are listed in the continuation of box C.

☒ Patent family members are listed in annex.

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Date of the actual completion of the international search

15 February 1994

Date of mailing of the international search report

22 Feb 94

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INTERNATIONAL SEARCH REPORT

Intern. Application No
PCT/US 93/10110

C.(Continuation) DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
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A	US,A,5 100 684 (MAGDA EL-NOKALY ET AL.) 31 March 1992 see column 1, line 6 - line 11 see column 4, line 13 - line 18 ----	1,6
A	EP,A,0 443 572 (TAKEDA CHEMICAL INDUSTRIES, LTD.) 28 August 1991 see page 4, line 57 - page 5, line 7 see claims 1,5,15-18 ----	1
A	US,A,3 751 265 (PAUL SEIDEN) 7 August 1973 see column 3, line 44 - column 4, line 6 see claim 1 ----	1-7
A	US,A,3 637 774 (VIGEN K. BABAYAN ET AL.) 25 January 1972 cited in the application see column 1, line 29 - line 31 see column 4, line 34 - column 7, line 74 -----	1-7

INTERNATIONAL SEARCH REPORT

Information on patent family members

International Application No

PCT/US 93/10110

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WO-A-9110368	25-07-91	EP-A- 0510070 JP-T- 5504062	28-10-92 01-07-93
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US-A-3637774	25-01-72	NONE	

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